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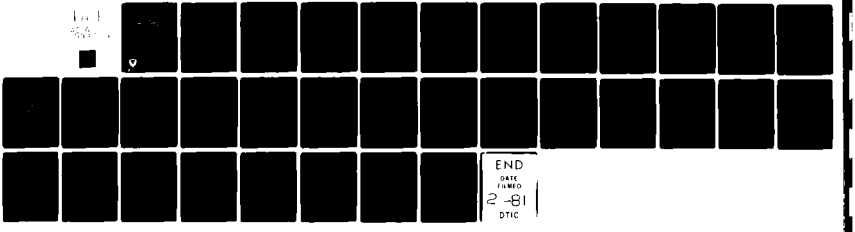

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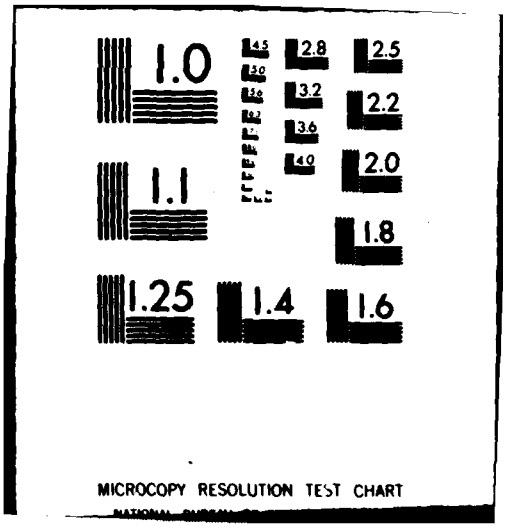
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INITIAL FIELD MEASUREMENTS OF ATMOSPHERIC ABSORPTION AT 9μM TO 11μM WAVELENGTHS

OCTOBER 1980

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A field-adapted spectrophone system employing a tunable CO_2 laser source (over wavelengths 9.2μm to 10.8μm) was used to measure atmospheric gaseous and particulate absorption at an isolated desert southwestern location in the United States. Measurements were made both for ambient conditions (when aerosol particulate absorption was negligible compared to that of gases) and for dusty conditions resulting from vehicular traffic. For ambient conditions,

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20. ABSTRACT (cont)

the gaseous absorption coefficient varied with time from expected levels upward by as much as a factor of 3. Sources which could be correlated with increased absorption are discussed. For dusty conditions the spectrophone data were compared with estimates of the absorption coefficient calculated on the basis of measured particle size distributions together with estimates of particle complex indices of refraction. Temporal variation of the absorption coefficient correlated closely for the two methods, while the calculated values were generally higher. Sampling and calculational uncertainties are suggested as likely to be responsible for this discrepancy.

SUMMARY

Measurements of both gaseous and aerosol atmospheric absorption made with a differential CO_2 laser spectrophone system have been reported. The spectrophone allows in situ measurements to be made on a real-time basis at frequencies of electro-optical systems and obviates some uncertainties present in other methods.

In the case of atmospheric gases, the use of this system shows how easily absorption may be significantly increased due to local and often innocuous appearing sources. Numerous trace gas pollutants at the 1 ppm level have absorption coefficients comparable to the ambient absorption and distinctive spectral patterns for the 9.2μm to 10.8μm emission line spectrum of a $C^{12}O_2$ laser. These factors suggest that the spectrophone device may be well-suited for air pollution studies.

Measurement of absorption by atmospheric particulates is more difficult than for gases. While the interaction between the spectrophone laser beam and the particles and the acoustical sensing are reasonably well understood, accurate sampling of all sizes of particles under windy conditions is difficult. (However, sampling particles with any other currently available technique faces similar difficulties.) One aspect of the importance of the particulate component is that of its extreme variability. Quiescent and low-wind conditions generally produce absorption coefficients that are negligible compared with the gaseous component at wavelengths around 10μm while high winds and/or anthropogenic disturbances can raise the particulate absorption by orders of magnitude well above the gaseous component.

ACKNOWLEDGMENT

The authors acknowledge the x-ray analysis performed by J. Whittler of the White Sands Missile Range Chemistry Laboratory and the aid of Mr. Glenn Hoidale of this laboratory for his many suggestions made during the preparation of this report.

CONTENTS

INTRODUCTION

Measurement of the absorption due to atmospheric gaseous and particulate matter at infrared wavelengths is a subject of current interest because of the relevance to communications, atmospheric sensing, and radiative transfer problems. Thus far, estimates of atmospheric absorption generally have been derived from measurements/estimates of the concentration of significant contributors at the wavelengths of interest. These concentration data are then converted to absorption coefficients at specific wavelengths. For gaseous absorption this may involve an insufficient data base of absorption coefficients, primarily for trace gases. In the case of particles, assumptions involving both shapes and complex indices of refraction must generally be made.[1]

This report discusses the relatively direct in situ field measurements of both gaseous and particulate absorption made by using a CO_2 laser spectrophone system. The spectrophone technique obviates many of the problems associated with much less direct approaches currently used for obtaining atmospheric absorption. The measurements were made in the spring of 1978 at a remote desert site in the White Sands Missile Range (WSMR), New Mexico. Although both gaseous and particulate absorptions are discussed, the focus is on absorption by soil-derived dust generated by vehicular traffic.

BACKGROUND

In a previous paper[2] a spectrophone for in situ measurements of atmospheric gaseous and/or particulate absorption was described. Environmental chamber measurements on known atmospheric aerosols (quartz and calcite dust) were made with this system which allows continuous flow through sampling and a high degree of isolation from acoustic noise. Measurements for known atmospheric gaseous absorbers (methane,[3] ammonia,[4] and water vapor[5]) have also been

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[1]S. G. Jennings, R. G. Pinnick, and H. J. Auvermann, 1978, "Effects of Particulate Complex Refractive Index and Particle Size Distribution Variations on Atmospheric Extinction and Absorption for Visible through Middle-IR Wavelengths," Appl Opt, 17:3922-3928

[2]C. W. Bruce and R. G. Pinnick, 1977, "In-Situ Measurements of Aerosol Absorption with a Resonant CW Laser Spectrophone," J Appl Opt, 16:1762-1765

[3]C. W. Bruce et al, 1976, "Application of Pulsed-Source Spectrophone to Absorption by Methane at DF Laser Wavelengths," Appl Opt Letters, 15:2970-2972

[4]R. J. Brewer and C. W. Bruce, 1978, "Photoacoustic Spectroscopy of NH_3 at the 9μm and 10μm $12C^{16}O_2$ Laser Wavelengths," J Appl Opt, 17:3746-3749

[5]K. O. White et al, 1978, "Water Vapor Continuum Absorption in the 3.5-4.0 μm Region," Appl Opt, 17:2711-2720

made with similar spectrophones. In general, the gaseous absorption measurements were in good agreement with those obtained by using long path transmission cells with beam folding optics (White cells). In the case of aerosols, the measurements were compared to Mie theory predictions of absorption (for spherical homogeneous particles) based on measured particle size distributions and knowledge of particle complex refractive indices (which had previously been measured). Though the particles were quite irregular in shape, the measured and calculated results compared reasonably well (generally within a factor of 2) for the substances measured.

However, when atmospheric gases and dust are encountered in the field, as they were during this research, the presence of unknown trace gases and particles of undefined composition complicates any comparison of spectrophone measured absorption as compared with that predicted. For gases, comparison presumes distinctive (and well-known) spectral patterns at the probe laser wavelengths to permit identification of the species. The particulate comparison is more complex. In this case, both elements of the comparison are based on measurements which may involve sampling errors which are hard to define; for example, they are a function of windspeed and wind direction. Spectrophone operational parameters (chopping rates and power densities) must be chosen to ensure adequate response to the particles. Principal criteria for these choices were that the period of optical beam modulation exceed the thermal cooling time for the largest contributing absorbing particles and that probe beam power densities be low enough that optical properties not be significantly altered for contributing sizes of particles of a given complex index and thermal diffusivity. Since a light scattering counter was used to measure particle size and concentration, its response and size resolution for particles characteristic of soil-derived dust must be defined. Finally, dust composition must be determined. Additional complications arise from the fact that the dust composition can be a strong function of size[6] (but unlikely in this case) and that refractive index information for the various dust constituents (which are required for Mie calculations of absorption) have been reported for only a few specific minerals.

MEASUREMENT SYSTEM

The ensemble of instrumentation consists of a spectrophone system for gaseous and particulate absorption measurements and correlative measurement instrumentation including a light scattering aerosol counter, a filter sampler, and a dew point hygrometer.

A schematic representation of the spectrophone system is shown in figure 1 where the laser beam path is traced through the alternate routes of spectrometer and spectrophones. Briefly, the differential resonant spectrophone system consists of two measurement elements. One samples both

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[6]J. D. Lindberg and J. B. Gillespie, 1977, "Relationship Between Particle Size and Imaginary Refractive Index in Atmospheric Dust," Appl Opt, 16:2628-2630

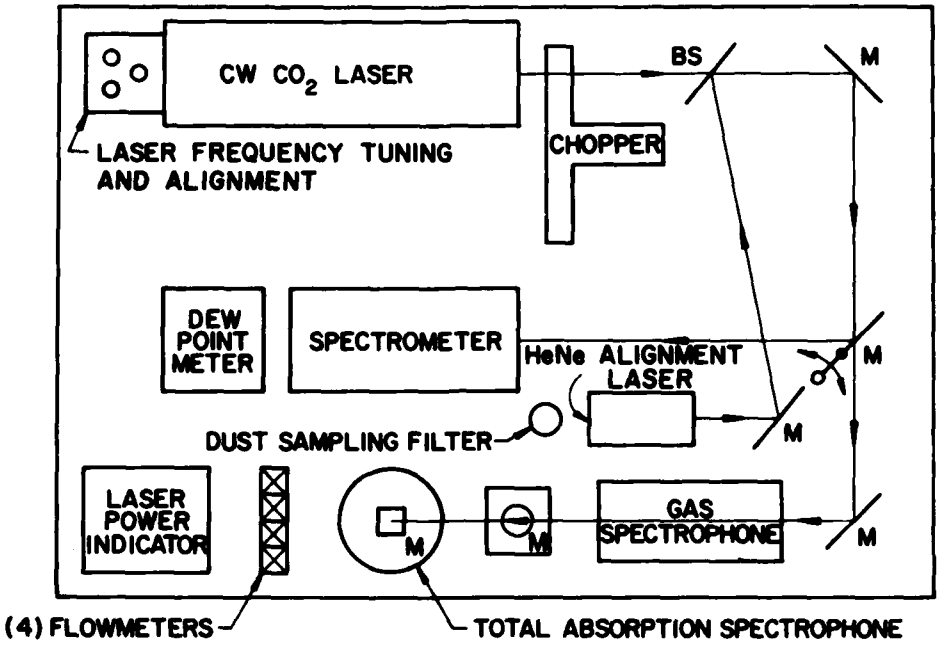

Figure 1. Schematic (top view) of the portable optical table showing optical paths for both the CO_2 and the He-Ne alignment lasers, spectrometer for laser line identification, gas and total absorption spectrometers, power meter, and two of the auxiliary systems used, i.e., (EG&G) dew point hygrometer and dust sampling filter using 0.2μm Nuclepore filter elements. Flowmeters are for the spectrophones and the auxiliary measurements. BS and M refer to adjacent beam splitters or mirrors.

gaseous and particulate atmospheric contents through an unfiltered intake; a second, whose intake is filtered to remove particles, samples only the gaseous component. The gain-equalized difference signal from the two elements is due to the particles, while switching the total absorption leg of the difference signal to electrical ground gives only the molecular absorption.

Both spectrophones are cylindrically symmetric with the coaxial laser probe beam. The gas spectrophone has cell end windows, while the beam and atmospheric constituents enter the "total" (gas and particle) absorption spectrophone through the open upper end as illustrated in figure 2. Acoustic wave filters and a microphone designed to promote laminar flow occupy the central region, followed by a tube with relatively high acoustical inertance terminating in a calorimeter. This tube is a low pass filter that minimizes flow noise from the pump connection (where it is not yet laminar) and any signal due to the interaction of the laser beam with the calorimeter. The CO_2 laser used for these measurements is a modified commercial unit (GTE Sylvania model 948) tunable from approximately 9.15μm to 10.8μm in about 80 lines. Signal processing for the spectrophone system is indicated in the block diagram of figure 3. Pulsed laser sources could be substituted for the CW source and signal processing as described previously.[5]

Considerable attention was given to the calibration of the light scattering counter (a Knollenberg model CSASP-100) used for measurement of aerosol size distributions and concentrations. The principle of operation of this instrument and determination of its response to spherical and nonspherical particles of various compositions are described in earlier papers.[7,8] Briefly, the instrument works on the principle that as aerosol flows through an illuminated volume, light scattered by a single particle into a given (near forward scattering) solid angle is measured and used to determine particle size by electronically classifying response pulses according to their magnitude. To define the instrument's size resolution for measurement of soil-derived aerosols, we have measured its response to uniform irregular particles with refractive indices characteristic of those of soil dust constituents. The results, which are shown compared to theoretical response calculations for spheres of equal cross section in figure 4, show that the instrumental size resolution is defined by an envelope enclosing the theoretical response curves. This envelope (shown by the dashed curves in figure 4) indicates what uncertainty in particle size results from a certain response (or pulse height) measurement, considering the fact that the

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[5]C. W. Bruce et al, 1976, "Application of Pulsed-Source Spectrophone to Absorption by Methane at DF Laser Wavelengths," Appl Opt Letters, 15:2970-2972

[7]R. G. Pinnick and H. J. Auvermann, 1979, "Response Characteristics of Knollenberg Light-Scattering Aerosol Counters," J Aerosol Sci, 10:55-74

[8]R. G. Pinnick and J. M. Rosen, 1979, "Response of Knollenberg Light-Scattering Counters to Non-spherical Poublet Polystyrene Latex Aerosols," J Aerosol Sci, 10:533-538

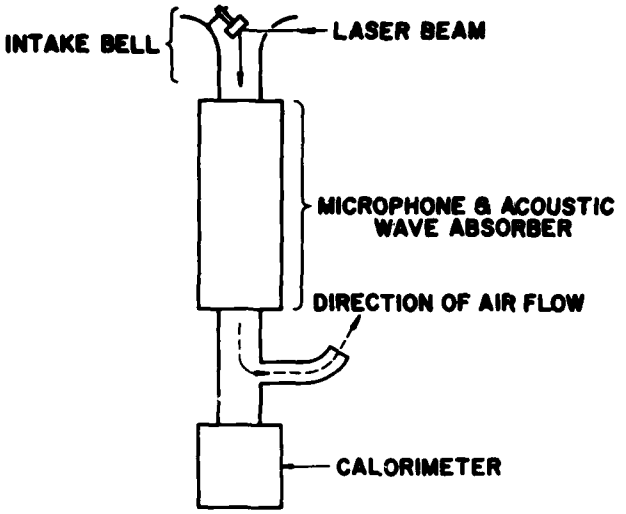

Figure 2. Schematic diagram (side view) of the total (gas and particle) absorption spectrophone.

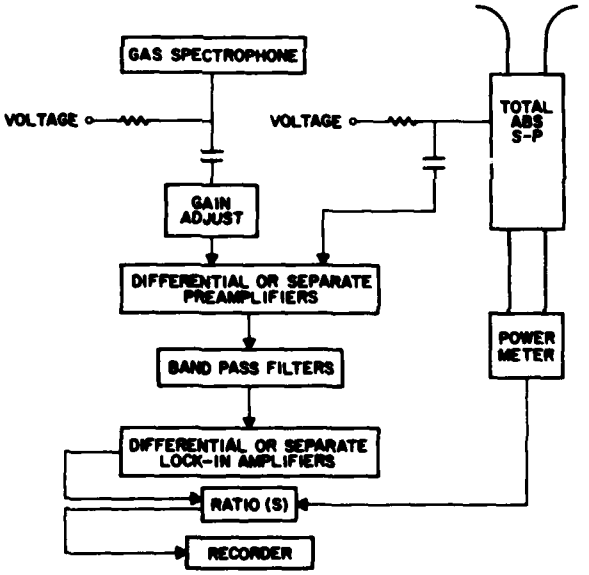

Figure 3. Signal processing for the CW laser resonant differential spectrophone system.

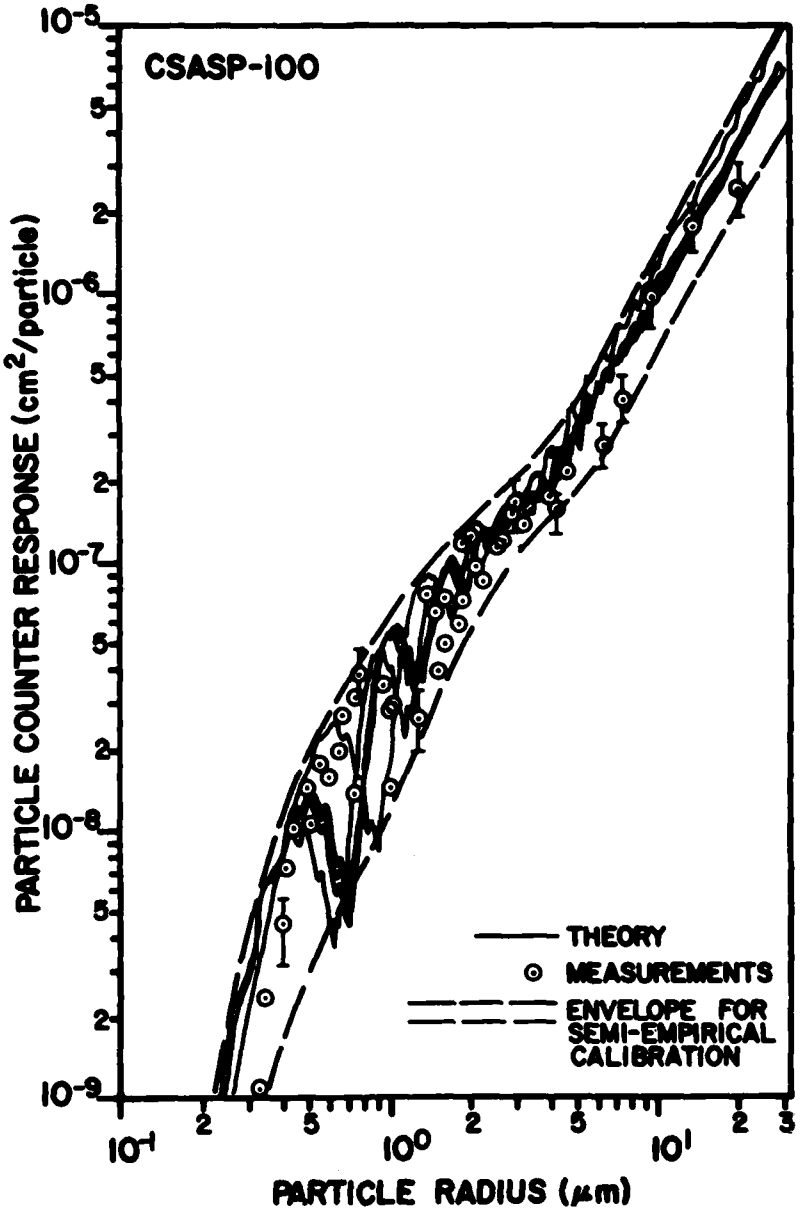

Figure 4. Knollenberg CSASP response: measured (circles) for nonspherical particles of doublet-shaped polystyrene (refractive index m = 1.592-0i), cubical sodium chloride (m = 1.54-0i), ellipsoidal potassium chlorate (m = 1.409-0i), and slightly nonspherical pollens and spores (puff balls, lycopodium powder, paper mulberry, ragweed, sweet vernal, and pecan) (with m = 1.53-0i); and calculated by using Mie theory (smooth solid curves) for spheres of equal cross section and refractive index. The envelope indicated by the smooth dashed curves defines an estimate of range of uncertainty in particle size that results for a particular response measurement for particles of unknown shape and refractive index.

particles are irregular and have different compositions (and refractive indices). This envelope has been extended slightly for application to soil dust constituents in an attempt to consider their slightly wider range of refractive indices and more irregular shapes. A curve drawn through the center of the envelope is then taken as the calibration for the instrument (to connect instrument response to particle size), and the envelope itself is used to define the particle sizing errors.

Air flows into both spectrophone and particle counter through intake bells which attempt to give relatively smooth flow through their measurement region and approximately isokinetic sampling when windspeeds are small.

MEASUREMENTS AND ANALYSIS

The CO_2 laser spectrophone system utilized for measurement of gaseous and particulate absorption generally can adequately define the spectral dependence of the particulate absorption in the 9μm to 11μm region, even though there are gaps between the four laser bands within this region. Molecular constituents cannot be completely characterized with the laser source since atmospheric gaseous absorption lines are more numerous than the laser probe lines and effectively narrower than the laser line spacing. However, the fixed patterns of the constituent gases generally do have strong, spectrally distinct contrasts for the absorbing constituents. This may permit identification of absorbing gaseous concentrations by deconvolution of the spectra using self-consistency.[9]

The measurements were performed partly to survey absorption by ambient atmospheric gases at the desert site. For purposes of gaseous analysis, measurements were made for 60 to 80 easily obtained laser lines with an estimated probable single measurement accuracy of 15 percent. However, significant changes in the absorption can occur in less time than is required for a complete manual spectral scan (about 1 to 2 hours). Such changes alter the observed spectral pattern, and deconvolution of the results may not give accurate concentrations by self-consistency of the spectra. Use of selected sets of laser probe lines is much more satisfactory since the measurement period can be made smaller than that for atmospheric changes. The technique used for choosing a minimum number of spectral lines is discussed by Samuel et al.[9] Water vapor and carbon dioxide produce most of the ambient level atmospheric absorption in the 9μm to 11μm atmospheric transmission spectra "window." Absorption by atmospheric carbon dioxide measured with a $C^{12}O_2^{16}$ laser probe is an exception to the assumption of distinctive spectra. Here the band profile of the absorption coefficient peaks varies relatively slow with wavelength. Examples of distinctive trace gas spectra are those of ozone, which appeared at levels as high as 0.030 ppm based on coefficients by

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[9]C. Samuel, C. W. Bruce, and R. J. Brewer, 1978, Spectrophone Analysis of Gas Samples Obtained at Field Site, ASL-TR-0009, US Army Atmospheric Sciences Laboratory, White Sands Missile Range, NM

Patty,[10] and ammonia, which appeared at levels as high as 0.023 ppm based on coefficients by Brewer.[9]

For ambient atmospheric conditions, the spectrophone measured net absorption coefficients ranged from the expected ambient values to several times those values. The magnitude and temporal variation in the absorption coefficients during one 3-hour midday period for a number of CO_2 laser lines are shown in figure 5. As mentioned, these variations were larger than expected, as was the average level of the absorption. The major contribution was expected to stem from carbon dioxide at roughly 0.07 km^{-1} and water vapor at about 0.04 km^{-1} for the relative humidity encountered (about 3 torr). In previous samples of desert air analyzed in the laboratory, the 9μm to 11μm gaseous absorption coefficients were also often approximately twice the expected value.[9] In those cases, possible contamination of the stainless sample system was suspected; however, the flowing sample changed in a matter of seconds, making contamination highly unlikely. Of course, the presence of personnel may have contributed to the high absorption. This potential problem will be avoided in the future by the development of an automated laser tuning system.

To point out the caution needed in interpreting the source of absorption in spectrophone measurements, several observations regarding interaction between personnel in the locale and the measurements might be of interest.

At one point, the absorption signal rose off scale (roughly 3x previous value) and persisted over several spectral lines. Then it was noticed that a meteorological observer about 15 m upwind was carrying a lighted cigarette. After the cigarette was extinguished, the absorption returned to previous levels.

On another occasion a particle counter was being cleaned with Freon 22 and acetone about 40 m downwind (windspeed about 7 m s^{-1}). The absorption coefficient increased when the cleaning began and decreased when the cleaning stopped. That the increase in absorption was due to these cleaning agents was obvious from the already familiar spectral dependence of these laboratory solvents. The increase (labeled t_2) relative to a more typical result (labeled t_1) is shown in the inset in figure 5 where a segment of the CO_2 laser 10μm P series is repeated for the two conditions.

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[10] R. R. Patty et al, 1974, "CO_2 Laser Absorption Coefficients for Determining Ambient Levels of O_3, NH_3, and C_2H_4," Appl Opt, 13:2850-2854

[9] R. J. Brewer and C. W. Bruce, 1978, "Photoacoustic Spectroscopy of NH_3 at the 9μm and 10μm 12_C O_2 Laser Wavelengths," J Appl Opt, 17:3746-3749

[9] C. Samuel, C. W. Bruce, and R. J. Brewer, 1978, Spectrophone Analysis of Gas Samples Obtained at Field Site, ASL-TR-0009, US Army Atmospheric Sciences Laboratory, White Sands Missile Range, NM

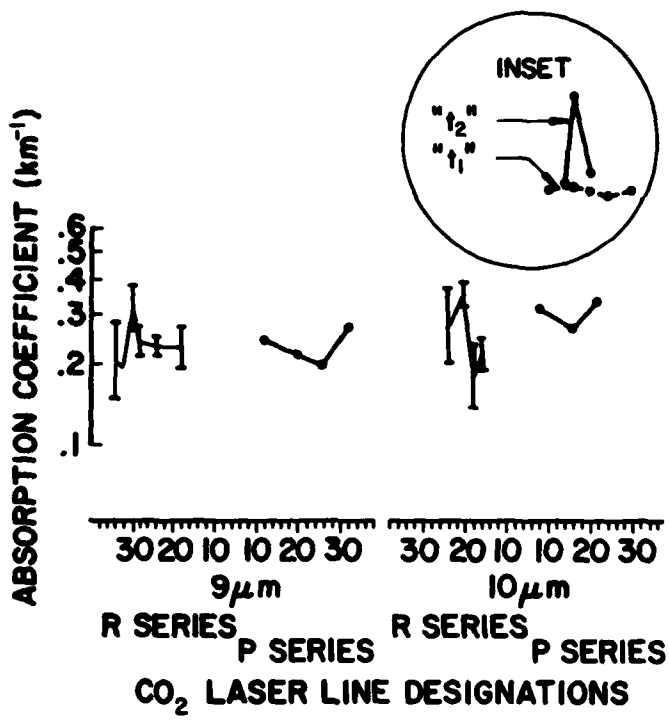

Figure 5. Atmospheric absorption coefficient data illustrating levels (points) and variations (vertical bar) for a 3-hour midday period (4 April 1978). Inset is a relative absorption (semilog) plot for 10μm P series CO_2 laser wavelength segment at two times whose significance is identified in the text. Straight lines connect measurement values for ease of identification.

A strong smell of creosote from a balloon enclosure persisted during some periods of high absorption, so an off-line spectrophone spectral analysis was performed in the laboratory on a sample taken from inside the enclosure. The results showed that creosote could not have been the source of the increase in absorption.

During measurements of atmospheric absorption under ambient conditions, the particulate absorption coefficient at 10μm was found to be generally less than 10 percent of the net gaseous absorption. Only occasional passage of vehicular traffic on the unpaved desert roads and one dust devil produced absorption coefficients larger than 10^{-1} km^{-1}.

Next, dust generated by vehicular traffic over the desert terrain was investigated. To correlate the spectrophone-measured absorption coefficient with an independent dust measurement, the (previously mentioned) Knollenberg probe was placed approximately 1 m from the spectrophone probe. The dust created by repeated circulation of a military truck vigorously driven over nearby desert terrain was then continuously measured with the spectrophone at a fixed CO_2 laser frequency while the counter accumulated data for consecutive 5-s intervals. The time resolution for the spectrophone was limited to about 1 s by the signal bandwidth used, and again the estimated probable error is 15 percent for particle radii less than 10μm (except for the elusive sampling errors).

Comparison of spectrophone measurements of absorption coefficient with calculations based on the Knollenberg data was then attempted. This comparison goes well beyond correlation of absorption coefficient with particle densities and requires some knowledge of the dust particle composition (or refractive index). Therefore, following the tests, vehicular dust which settled onto the ground was taken to the laboratory for spectral and chemical analysis.

The spectral analysis was accomplished by dispersing the sample dust in an environmental chamber and scanning the spectral region of concern using spectrophone systems similar to those used in the field. The reliability of these results depends to some degree on the assumption that the size distribution of redispersed dust samples is the same as it was in the field.[1]

The chemical analysis was attempted by using two methods: x-ray diffractometry and infrared spectrophotometric analyses. The x-ray analysis revealed a strong quartz component but little else since not all of the dust consisted of crystalline material. The infrared spectrophotometric analysis was much more informative because of the distinct infrared spectral absorption

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[1]S. G. Jennings, R. G. Pinnick, and H. J. Auvermann, 1978, "Effects of Particulate Complex Refractive Index and Particle Size Distribution Variations on Atmospheric Extinction and Absorption for Visible through Middle-IR Wavelengths," Appl Opt, 17:3922-3928

patterns of the minerals involved.[11] In this analysis, the sample transmission spectra between wavelengths 2.5μm to 40μm were measured with a Perkin Elmer model 521 infrared grating spectrophotometer. The spectral transmission "fingerprints" of the substance under test were compared with those of known pure reference samples to assess the fractional composition of the unknown dust sample.[12] In this spectrometric estimate of mineral composition, the following assumptions were made: (1) the sample and reference particulate size spectra are identical functions of those distributions, (2) the absorption resonance peaks were assumed to be unique to a particular mineral, and (3) sample and reference minerals were assumed to be in the same chemical state. Under these assumptions the fractional mineral composition of the dust sample was then calculated by converting the transmission spectra to absorption coefficients and scaling the absorption resonance peaks linearly with mineral mass concentration. The results of this analysis are shown in table 1. It is noteworthy that the minerals identified (quartz, montmorillonite, calcite, and gypsum) were also found in naturally occurring aerosol samples collected within 4 km of where this test was conducted, although the proportions of each mineral were markedly different.[11]

TABLE 1. RESULTS OF SOIL ANALYSIS WITH COMPLEX INDICES

Mineral	Identification Wavelength (μm)	Fractional Composition (%)	Complex Index at 9.55μm (n_r, n_i)
Gypsum	2.82	26	2.0, 0.30
Calcite	6.94	14	1.7, 1.20
Montmorillonite	9.80	35	0.86, 1.28
Quartz	12.82	25	1.0, 5.4

The fractional composition values of table 1 and the complex refractive indices of the constituents, with the Lorenz-Mie theory, can be used to

[11]G. B. Hoidale and A. J. Blanco, 1969, "Infrared Absorption Spectra of Atmospheric Dust over an Interior Desert Basin," Pure and Appl Geophys, 74:151-164

[12]G. Duyckaerts, 1959, "The Infra-Red Analysis of Solid Substances," Analyst, 84:201-214

calculate the contributions to the total absorption coefficient of each component. Assumptions made in this process are those of particle sphericity, homogeneity, and similar size distributions for each constituent. In addition, for the birefrigent quartz and calcite particles, the absorption was calculated by considering two populations of particles--one having refractive index of the ordinary ray and one the extraordinary ray--and adding the absorption coefficients for these fractional populations on a 2/3 to 1/3 basis.

Presently, even a rough estimate of the effect of these assumptions on the calculated absorption coefficients would be very difficult. Furthermore, the complex indices for these constituents are not well-known and montmorillonite (for example) does not have a fixed composition. Quartz is probably the best defined with respect to index; the others may be accurate within perhaps a factor of 2.

Figure 6 shows an example of the spectrophone measured dust absorption coefficients compared to those predicted from the Knollenberg counter dust size distribution measurements together with the spectrophotometric analysis of the dust composition. Knollenberg data were taken continuously during this 2-minute period, although the instrument particle count rate exceeded the maximum reliable rate (determined in the laboratory to be about 1000 s^{-1} under these conditions) for several 5-s accumulation intervals. (The predictions of absorption for this suspect data generally fall above the 100 km^{-1} level in figure 6.) In any case, the predicted absorption for all of the Knollenberg data displays similar temporal variation as the spectrophone measured absorption, although the values are higher by a factor of 3 to 5. The "error" bars shown for the Knollenberg-based predictions indicate only possible error caused by uncertainty in particle size determination made from the counter measurements; no estimate of errors accompanying the assumption of spherical particles, of the approximate treatment for birefringent particles, and of the assumption of similar size distribution for each dust mineral constituent was attempted.

Thus, in view of the multitude of rather critical assumptions made in predicting absorption from the Knollenberg size distribution data, and considering that the aerosol sampling losses may be significant and different for the spectrophone and Knollenberg sensors, agreement of the measured and predicted absorption in figure 6 is considered respectable.

To determine how much absorption (at the 9.21μm wavelength) is contributed by particles of various sizes the differential absorption coefficients were calculated from the Knollenberg size distribution measurements. The results for 35 s of data (corresponding to times denoted a-g in figure 6) are presented in figure 7. These results suggest that the absorption is dominated by particles with radii in the 1μm to 5μm range regardless of dust loading.

Spectrophone measurements of the spectral dependence of the absorption for the vehicular dust redispersed in a laboratory environmental chamber are shown in figure 8. Because of the uncertainties mentioned earlier for the complex indices of the dust constituents, no attempt was made to calculate a result

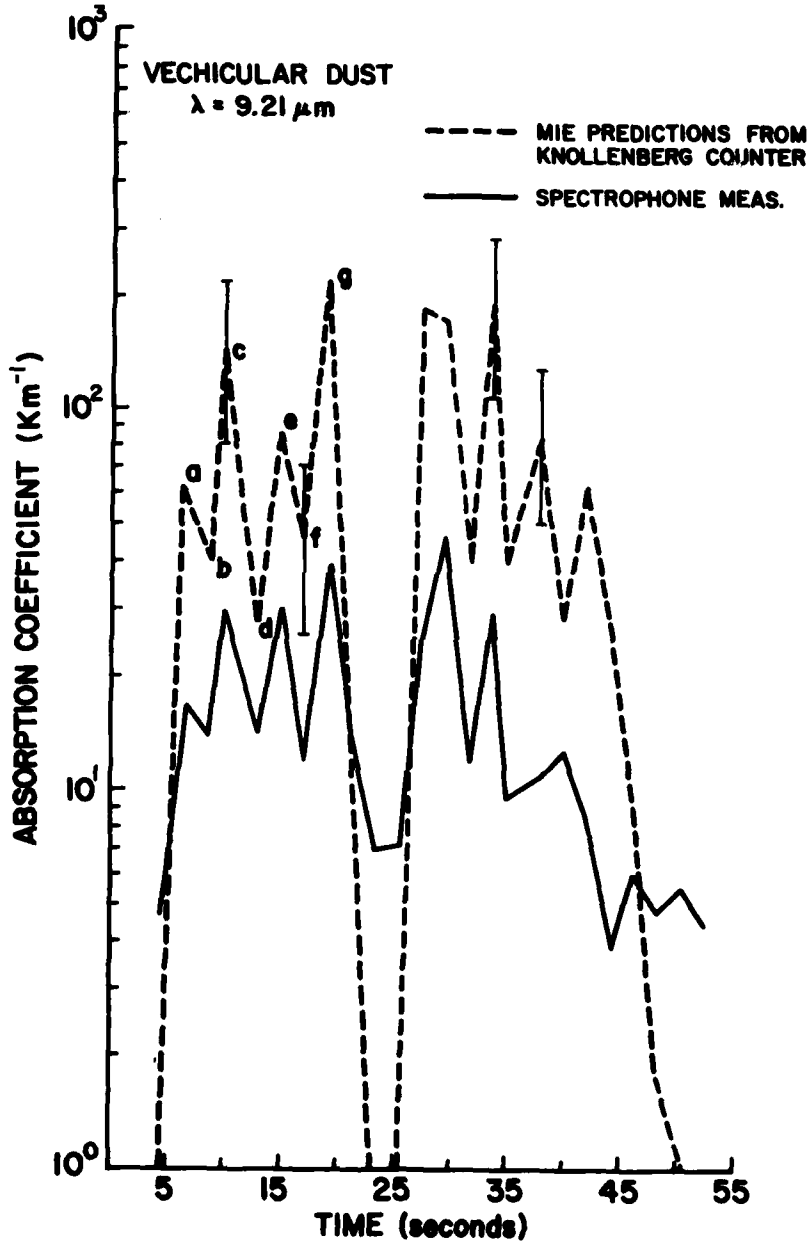

Figure 6. Absorption coefficient of vehicle dust at the 9.21μm, R(34) CO_2 laser line as a function of time during the test: measured with in situ spectrophone (solid line); and calculated from particle size distribution measurements made with a Knollenberg light scattering aerosol counter (dashed line). The "error" bars superimposed on the Knollenberg-based predictions indicate only the error caused by uncertainty in particle size determination. Other sources of error in the predictions (see text) and differences in the spectrophone and Knollenberg probe sampling efficiencies are expected to cause the remaining discrepancy between measured and predicted absorption.

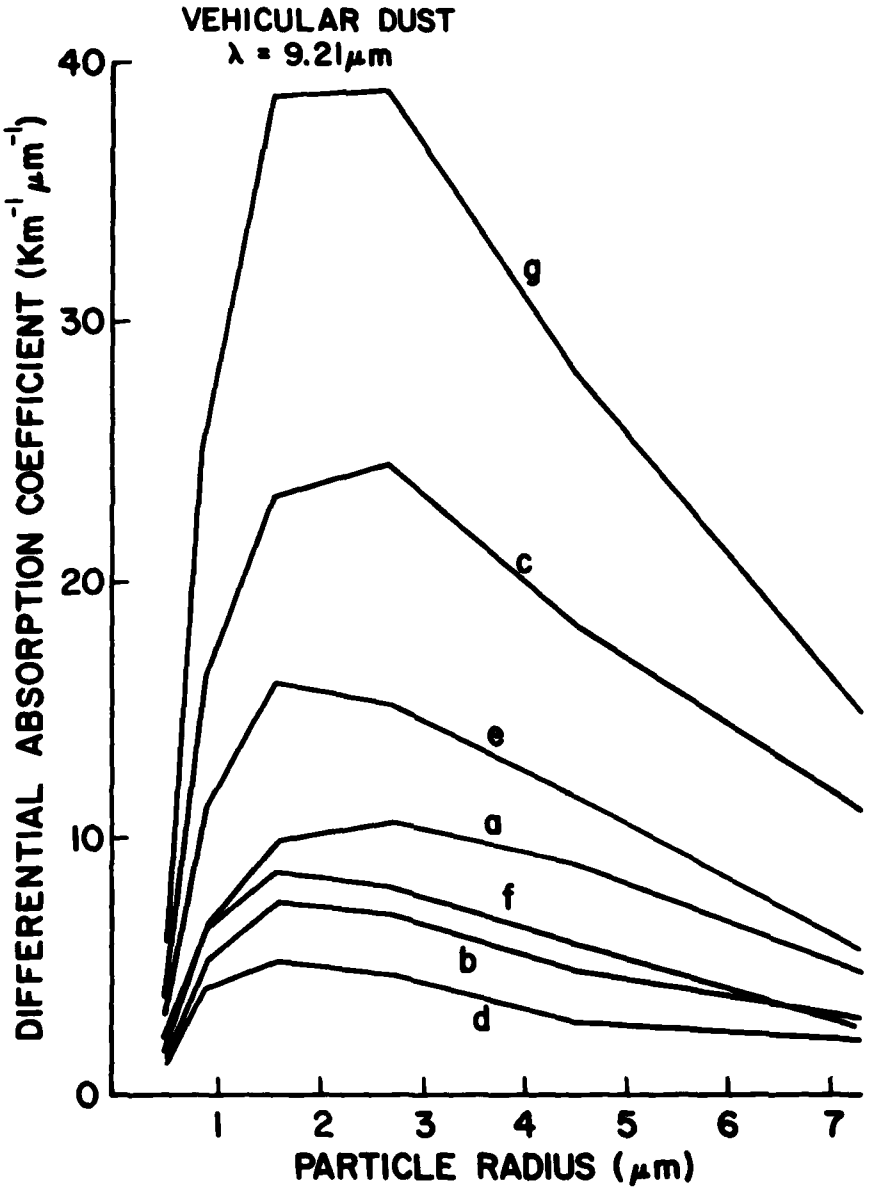

Figure 7. Differential absorption coefficients of vehicular dust showing that 1μm to 5μm (equivalent) radius particles dominate absorption at the 9.21μm CO_2 laser line. These coefficients are calculated from the Knollenberg size distribution measurements made at times a-g (in figure 6) together with estimates of the vehicular dust refractive indices (see text for additional details of this calculation).

for comparison. However, the spectral profiles for quartz and calcite dusts measured with spectrophones (for similar size distributions) and transmission spectra for montmorillonite and gypsum measured with the spectrophotometer techniques suggest that the strong absorption feature in the 9μm region in figure 8 is due partly to quartz, but that its broad character and its extension past 9.6μm is probably due to the presence of clay minerals such as montmorillonite.

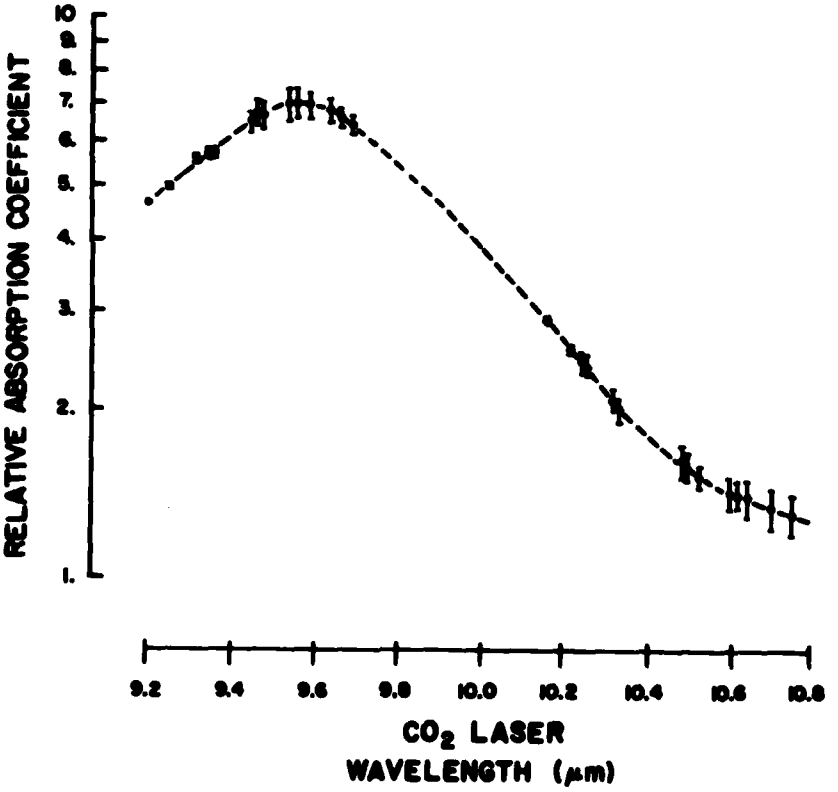

Figure 8. Spectrophone absorption measurements of the spectral dependence of samples of vehicle dust.

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